

17005 2

# **FIELD SAMPLING PLAN**

**SITE PREPARATION AND MATERIAL REMOVAL**

**FINAL DESIGN  
ENVIRO-CHEM SUPERFUND SITE  
ZIONSVILLE, INDIANA**

**Prepared For:  
ENVIRONMENTAL CONSERVATION AND  
CHEMICAL CORPORATION TRUST**

**Prepared By:  
AWD TECHNOLOGIES, INC.  
INDIANAPOLIS, INDIANA**

**AWD PROJECT NUMBER 2259**

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### **NOTICE**

This document is a portion of the overall design package and, therefore, cannot be referenced, in whole or in part, as a standalone document for any other purpose.

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## **1.0 INTRODUCTION**

This Field Sampling Plan (FSP) has been developed and is being submitted as a Final (100%) Design for the Site Preparation and Material Removal (SPMR) phase of the Remedial Actions to be conducted at the Environmental Conservation and Chemical Corporation Site (ECC Site), located in Zionsville, Indiana.

ERM-North Central has previously submitted a number of versions of a two-part Sampling and Analysis Plan for the ECC Site which contained a Part I - Field Sampling Plan and a Part II - Quality Assurance Project Plan. The Sampling and Analysis Plan addressed site preparation, material removal and remedial action activities, although the plan primarily focused on remedial action activities.

The previous ERM-North Central submittals of the Sampling and Analysis Plans and the corresponding U.S. EPA Region V comments are as follows:

1. Sampling and Analysis Plan, Revision 0, March 1, 1989
2. Sampling and Analysis Plan, Revision 1, December 10, 1991
3. U.S. EPA Region V Comments on Revision 1, February 21, 1992
4. Sampling and Analysis Plan, Revision 2, March 24, 1992

AWD Technologies, Inc. (AWD) has revised the ERM-North Central Sampling and Analysis Plan, Revision 2, to further address the U.S. EPA comments. The previous Sampling and Analysis Plan two-part format has been modified to include the Field Sampling Plan as part of the Quality Assurance Project Plans. The Sampling and Analysis Plan terminology is not used in the AWD plans.

The Final Design for the ECC Site has been further modified to include two design packages: (1) Site Preparation and Material Removal and (2) Remedial Action. The Site Preparation and Material Removal phase includes preparation of the support zone and removal of above ground tanks, buildings, and miscellaneous debris. The Remedial Action phase includes in-situ soil treatment by soil vapor extraction, capping of the soil treatment area, and verification and compliance monitoring.

This FSP is intended to cover all necessary sampling and analytical procedures to be implemented during preparation of the site when removal of obstructing and miscellaneous materials and debris will occur. This FSP is designed to provide adequate classification and profiling of the materials listed in Appendix A of the Contract Technical Specifications in order to satisfy acceptance criteria for offsite disposal facilities.

## **2.0 PROJECT DESCRIPTION**

### **2.1 Site Location**

The ECC Site is located in a rural area of Boone County, about 5 miles north of Zionsville and 10 miles northwest of Indianapolis, Indiana (Figures 2-1 and 2-2).

### **2.2 Site Description**

The Site is defined as the area bounded by the proposed perimeter fence, which includes the 3.053-acre remedial boundary the support zone, and the buffer zone between the proposed fence and the north and eastern sides of the Site. A buffer zone on the southern side of the Site contains a proposed Remedial Contractor equipment laydown area.

Directly west of the Site is an active commercial waste handling and recycling facility operated by the Boone County Resource Recovery Systems, Inc. (BCRRS). Access to the Site will be from State Route 421 and will be shared with BCRRS.

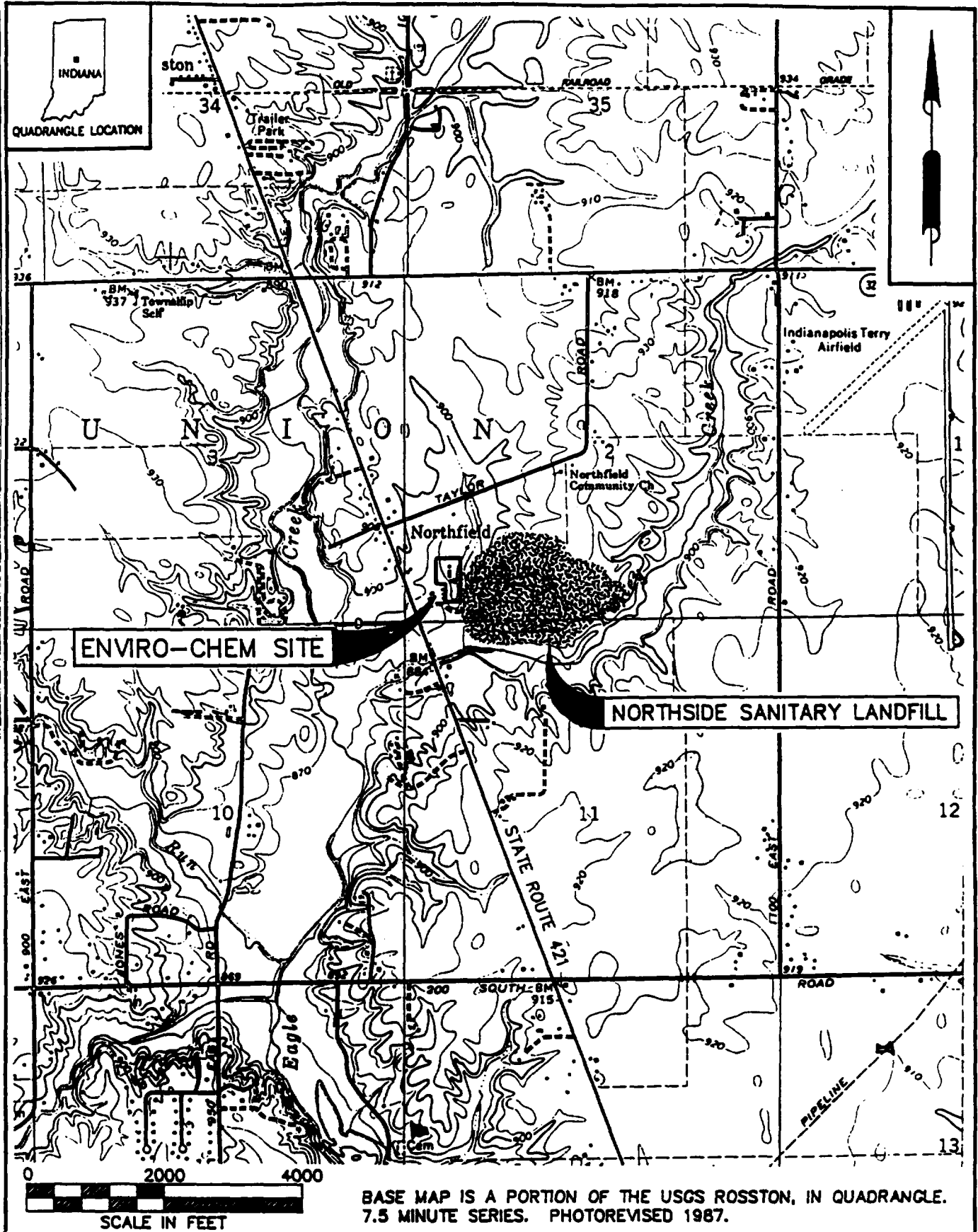
Directly east of the Site across an unnamed ditch is the inactive Northside Sanitary Landfill (NSL) landfill. This facility is also a Superfund Site and is presently undergoing remedial design activities. The south end of the Site is approximately 500 feet from an existing residence and is approximately 400 feet from Finley Creek, the main surface water drainage in the site area.

Residential properties are also located to the north and west, within 1/2 mile of the facilities. A small residential community, Northfield, is located north of the Site on State Route 421. Approximately 50 residences are located within 1 mile of the sites.

The Site is in an area that is gently sloping, predominantly to the east toward the unnamed ditch. The unnamed ditch runs north to south along the eastern edge of the Site and drains the Site either directly or from tributary ditches on the north and south ends of the Site. The unnamed ditch flows south from the Site to Finley Creek.







Various solid waste materials are present at the Site both within the remedial boundary and within the support zone. Emergency actions undertaken prior to 1990 have resulted in the removal of the major sources of contamination. The materials at the Site include cleaned tanks, the process building, the A-frame structure, the concrete pad with approximately 250 drums, and miscellaneous debris.

### **2.3 Summary of Work**

The Site Preparation and Material Removal phase includes the following:

- Preparation of a site support zone which will consist of facilities to support the materials removal efforts and subsequent corrective actions; placement of temporary controls; and design and layout of ingress, egress (personnel and traffic), and materials handling and storage areas.
- Remove physical obstructions including tanks, buildings, debris, and any other above ground obstructions prior to initiation of remedial design construction.

Sampling and analyses will be performed on selected materials for removal based on visual classifications, field screening, and RCRA waste characterization.

### **3.0 FIELD SAMPLING PLAN OBJECTIVES**

#### **3.1 Objectives**

The objectives of the FSP are to:

1. Describe applicable procedures for the collection of representative samples from waste and debris for subsequent characterization and offsite disposal.
2. To assure that samples are collected in a fashion that will provide the highest level of confidence in subsequent testing and results so that material waste and debris can be directed toward appropriate disposition.

## **4.0 MATERIAL AND DEBRIS HANDLING AND STAGING**

### **4.1 General**

Items which will be handled during the SPMR phase of the site remedy will fall into three general categories of materials including (1) RCRA regulated hazardous waste, (2) special waste, and (3) excluded materials including salvage material. In accordance with state regulations for the purposes of SPMR and meetings with IDEM, not all debris will be handled as special waste which is outlined within 329 IAC 2-21.

The following is a breakdown on how items will be handled and removed (disposed) from the site as discussed and agreed upon with IDEM during the design effort.

### **4.2 Tanks**

Presently 53 used process tanks are staged on the west side of the ECC property (Appendix A, Table 1). Additionally, there are a few smaller volume fuel tanks which are among the building and outside debris. The old process tanks will be handled according to Section 02081 - Tanks and Figure 1 in Appendix C of the Site Preparation and Material Removal Technical Specifications. The miscellaneous small fuel tanks will be checked for any content, and the content removed and staged for sampling if required.

IDEM agrees that salvage of metal and salvageable materials is the best final disposition of this material. Salvageable metals that can be decontaminated, including such items as the cut up tanks may be salvaged with no formal notice or approval from IDEM required. All materials slated for salvage will be decontaminated and decontamination records maintained.

### **4.3 Bulked Liquids**

Onsite bulking of liquid waste will be the greatest volume for handling and disposal considerations. Liquid waste will originate from SPMR decontamination activities and liquids pumped from other onsite vessels and structures being removed. Liquids will continually be bulked in an onsite liquid hazardous waste tanker supplied by the liquid treatment facility or other licensed general hauler. Initial profiling should be completed using the chemical

information presented in Table 1-1 of the SPMR QAPP since it is anticipated that accumulated waters within onsite features and decontamination waters will present no great deviation in characteristics or concentration from those ranges recognized from the Remedial Investigation.

Sampling of the hazardous waste tanker may be required by the TSD facility per load for verification. When it is suspected that decontamination activities may produce wastewater which would alter the composition of the bulked liquids then a sample will be required for laboratory characterization prior to bulking. These waters will be held separately until found compatible with the tanker liquids and/or acceptable to the liquid waste treatment facility.

#### **4.4 Process Building**

Tables 3 through 6 in Appendix A show the materials and debris which exist inside the onsite buildings, in miscellaneous debris areas, and associated with past investigative activities. Most of the materials and debris are anticipated to be disposed of as solid nonhazardous waste, or salvaged and/or recycled.

The non-metallic materials which make up the process building (i.e., block, roofing materials, wood, etc.) will be handled according to the following:

- Block, brick, concrete, wood, and miscellaneous materials associated with the old process building will be sampled by compositing similar materials and analyzing them for RCRA toxicity characteristics. Analytical results will be submitted to the Indiana Department of Environmental Management (IDEM) Special Waste section for anticipated one-time disposal approval into an IDEM permitted waste landfill approved for acceptance of special waste.
- The large boiler within room 1 of the process building will be handled as hazardous waste and disposed of accordingly. Prior to removal of the boiler, the insulation materials and brick within the boiler shall be sampled and analyzed to confirm the absence of asbestos. "Grab" samples of the insulation materials and brick shall be collected by drilling, chipping, or cutting these materials as necessary to obtain a suitable sample volume for preparation of the composite samples.

#### **4.5 Miscellaneous**

Certain items such as herbicides, pesticides, paints, etc. shall be placed in laboratory packs and placed on the southern concrete pad pending laboratory analysis for disposal. Pesticides and herbicides shall be disposed of in accordance with the requirements of the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA). Samples from these items may be required, and will be coordinated between the Remedial Contractor and the TSD facility for sampling and profiling.

Removal of the transformer by the local utility company for reuse or recycling is allowable. The utility (or whomever removes the transformer from the pole) must follow the contractor's Health and Safety Plan when going onto the site.

Non-leaking fluorescent light ballasts may be disposed of as general solid waste in groups of 25 or less at a time. Multiple shipments of 25 or fewer ballasts is acceptable to meet this requirement. If more than 25 ballasts are disposed of in one shipment, special waste approval must be obtained from IDEM.

Any leaking fluorescent light ballasts containing PCBs must be disposed of in accordance with TSCA regulations or 329 IAC 4.

Fluorescent tubes must be handled as RCRA hazardous waste. IDEM has historical information that 50 percent of fluorescent tubes tested have failed TCLP for Cd, Pb, and/or Hg.

If the Contractor has reason to believe that surface debris is contaminated when slated for disposal, Special Waste certification will be obtained prior to disposal. This will apply to materials that cannot be decontaminated, excluding wood, brick, etc.

Any material contaminated by listed hazardous waste which, when tested, has detectable hazardous waste constituents, will be handled as a hazardous waste for disposal (mixture rule).

## **5.0 SAMPLING EQUIPMENT AND PROCEDURES**

### **5.1 Bulked Liquids (Tanker)**

#### **5.1.1 Liquid Waste Tanker Sampling Equipment**

- Bailers
- Open tube samplers
- Pond samplers
- 250 ml glass beakers
- PVC pipe of sufficient strength
- Wrenches

#### **5.1.2 Procedures**

Field sampling procedures for collecting tanker content samples using an open tube sampler, pond sampler, or an open bucket sampler are as follows:

1. Gain access (e.g., steps, ladders, man-lift, etc.) to the tanker's top.
2. Slowly open release valve (if any) to bring the tanker to atmospheric pressure.
3. Loosen access port/cover bolts and remove port/cover.
4. If no access port/cover is available, unscrew cap of top opening.
5. Insert a decontaminated sampling device into tanker slowly to allow stratified content (if any) to fill the sampler. (Note: Samples will be collected at different horizontal and vertical points.)
6. Retrieve the sampling device and wipe it with a disposable absorbent pad (place the pad in appropriate container).
7. Transfer the sample(s) into appropriate containers.



8. Repeat Step 5 until enough sample volume is obtained, as required.
9. Cap the sample container tightly and place in container carrier, make sure sample has been labeled and identified.
10. Replace cap or access cover and secure.

If sample collection from the bottom valve is required, the following additional steps will be included:

1. Make sure that sampling is carried out on the wastewater storage pad.
2. Place sample container beneath the valve.
3. Open valve slowly to ensure a slow, controlled flow of material.
4. After obtaining enough material, close valve securely.
5. Cap the sample container tightly and place in container carrier. Make sure sample has been labeled and identified.
6. Check valve for any signs of leaking.

## **5.2 Building Materials (Structural Non-Metal)**

### **5.2.1 Structural Materials Sampling Equipment**

- Hammers
- Chisels
- Masonry saw and blades
- Masonry drills
- Ladders

### **5.2.2 Procedures**

This sampling approach must be submitted and reviewed by IDEM, and will consist of physically (i.e., drilling, hammering, cutting, etc.) "grab" sampling representative specimens of the structural nonmetallic portions of the building. This may include composite sampling of suspected or visually contaminated areas.

- Visually inspect process building masonry walls and other structures for visually contaminated surfaces. (It is anticipated that the boiler room may be an ideal area to conduct multiple sampling since it is recorded that solvents were burned here.)
- Plan out a representative sampling approach to any adjacent large areas stained or suspect.
- At least one sample per wall per room will be composited for subsequent analysis.
- Remove representative portions of cinder block, brick, concrete.
- Collect pieces which have been unaffected by the destructive sampling (unscarified surface).
- Place pieces into appropriate sample containers.

### **5.3 Other Sampling Equipment**

The following equipment may be used for some, if not all sampling activities:

- Vacuum pumps
- Tool box (miscellaneous tools)
- Sample containers
- Latex gloves
- Water (potable, distilled)
- Vermiculite (packing material)

- Sample labels
- Indelible markers
- Duct tape
- Plastic bags (trash, sandwich, Ziploc, etc)
- Clamps (stainless steel or Teflon)
- Rope, cord
- Paper towels
- Spatulas
- Brushes
- Paint cans (1 gallon, empty)
- Plastic sheeting (Visqueen)
- Sorbent pads
- Utility knife

#### **5.4 Other Sampling Activities**

The Sampling Team Leader will be responsible for recording all pertinent information into the sample logbook. At a minimum this will include the following:

- Sample location
- Sample number
- Material phase (i.e., solid, liquid, sludge, etc.)
- Sample time
- Sampler's initials
- Other important observations

The above is in addition to other entries made at the start of each work day. Once sampling has been completed in a particular building(s), the Sampling Team Leader will be responsible for delivering the samples to the sample receiving area at the decontamination pad. The Sampling Team Leader will then complete a chain-of-custody form and assist in readying the samples for shipment. This will involve documentation of sample numbers, date, time, and preservatives, as appropriate, as well as packing the "coolers" for shipment. Samples will be shipped on a daily basis so that analysis can be performed within required holding times.

## **5.5 Sample Frequency**

Sample frequency and quantities are presented in Table 7-1 of the SPMR QAPP. Most sampling frequencies will be a field determination by the Remedial Contractor based on the characteristics of the materials with respect to visual classification (drums), field organic vapor screening, and RCRA waste characteristics.

## **6.0 SAMPLING EQUIPMENT DECONTAMINATION**

### **6.1 General**

The following describes standard operating procedures for the decontamination of equipment and tools that may come into direct contact with a field sample intended for analytical analysis. This procedure only addresses the decontamination of equipment as it pertains to the chemical integrity of samples for analysis and is not intended for use in health and safety decontamination of personnel, materials, and equipment that may become contaminated during field operations.

### **6.2 Applicability**

Decontamination of all analytical devices, sampling tools, and storage equipment that may come into direct contact with a field sample are necessary in order to achieve analytical results that are representative of true field conditions.

The decontamination procedures below may be modified as long as the chemical integrity of the field sample is maintained within the analytical detection limits and the sample source is not permanently compromised. Anticipated contaminants and concentrations, media (water, soil, etc.), surface area of possible cross contamination, method of sampling, and many other factors will be considered when establishing a sampling equipment decontamination procedure. Any modification of the procedures below will be carefully thought out, approved by the Construction Manager, and documented accordingly.

### **6.3 Procedures**

All equipment will be considered contaminated unless determined otherwise. In order to provide consistency to the decontamination procedure, a designated sampling team crew member will be responsible for equipment decontamination. Similarly, it is desirable to decontaminate all the equipment necessary for a field task in the laboratory prior to mobilization. In this way, field decontamination will be limited.

### **6.3.1 Decontamination Equipment List**

The following equipment is needed for equipment decontamination:

- Clean disposable rubber gloves
- Wastewater container (drum)
- Clean water spraying device
- Clean brushes
- Plastic garbage bags
- Ten percent nitric acid solution in squirt bottle (squirt bottle is not recommended for transportation)
- Acetone or methanol in squirt bottle (squirt bottle is not recommended for transportation)
- Deionized/distilled water (DI water)
- Clean buckets and other containers, as needed (small plastic swimming pool)
- Plastic ground sheet (Visqueen)
- Aluminum foil
- Package labels and pen
- Potable water, warm if available
- Steam cleaner (optional)

### **6.3.2 General Equipment Decontamination Procedure**

The following steps will be considered the general equipment decontamination procedure:

- Cover hands with disposable rubber gloves.
- Wash and scrub as necessary with a solution of non-phosphate detergent and potable water (warm water if available). Thorough steam cleaning may be used as a substitute for this step.
- Rinse thoroughly with potable water (warm water if available).
- Rinse with 10 percent nitric acid solution.
- Rinse with DI water.
- Rinse with hexane or methanol.
- Rinse with DI water.
- Air dry.

The nitric acid rinse is only required if inorganic (i.e., metals and general chemistry parameters) analysis is intended for the sample. The solvent rinse is only required for organic analysis.

All waste liquids and solids generated by the decontamination procedure will be containerized and disposed of properly.

Decontaminated equipment not intended for immediate use may be placed in plastic bags and sealed. All handling of decontaminated equipment will be performed using disposable rubber gloves. Care will be exercised in the storage of decontaminated equipment. Sampling personnel will avoid solvents, greases, oils, gasoline, water, dusts, and other potential sources that might contaminate the equipment before use.

## **7.0 SAMPLE HANDLING AND TRACKING**

### **7.1 Sample Identification**

Each sample collected will be assigned a unique identification number and placed in an appropriate sample container. Each sample container will have a sample label affixed to the outside with the date, time of sample collection, site name, type of sample, and sampler's name recorded on the label. In addition, this label will contain the sample identification number, analysis required and chemical preservative added, if any. All documentation will be completed in waterproof ink.

The sample identification number will be a unique alphanumeric code which will identify the project site, sample location, sample type, and sample number. The sample ID for specific locations will have the following for group identifiers:

Site Code - Sample Location - Sample Type - Sample Number

The alphanumeric code for each sample will initiate with the three-letter project site code for the Environmental Conservation and Chemical Corporation Trust (ECC Trust). This will be followed by the sample locations which will be identified by a two-digit number corresponding to the inventory followed by an A1-6 if located in any of the debris areas.

The sample type identifiers will be as follows:

- PBM - Process Building Material
- TK - Tanker Content

For example, the first sample from the process building will be identified as:

ECC-04A1-PBM - 01



This is an optional identification tracking system, the Remedial Contractor may create a different approach which should be documented and approved by the Engineer. Movement of materials during segregation and staging would necessitate the updating of the inventory tables, if the above system is used.

## **7.2 Field Documentation**

Field notebooks will be maintained by the Sampling Team Leader to record all data collecting activities performed at the site. Entries will be as descriptive and detailed as necessary so that a particular situation can be reconstructed without reliance on the collector's memory. The cover of each book will contain the following information:

- Project name and number
- Project location
- Book number
- Activity type
- Start date
- Stop date

At a minimum, entries will consist of the following:

- Date
- Start date
- Weather
- Field personnel present
- Signature of the person making the entry
- Type of activity conducted
- Sampling location
- Sample identification number
- Description of depth of sampling point
- Type of sample (matrix)
- Pertinent field observations

All measurements made and samples collected will be recorded. All entries will be made in indelible ink. No erasures will be permitted. If an incorrect entry is made, the data will be crossed out with a single strike mark and initialed. Entries will be organized into easily understandable tables, if possible.

### **7.3 Chain-of-Custody**

To maintain and document sample possession, the following chain-of-custody procedures will be followed. A chain-of-custody record will be completed once the samples are brought to the on-site sample receiving area. This record will include, but not be limited to, the following information:

- Project name and number
- Name(s) of sampler
- Sample identification number and location
- Date and time of collection
- Number and type of containers
- Required analyses
- Preservatives
- Courier
- Signatures documenting change of sample custody

Chain-of-custody forms will accompany any and all samples which are shipped off-site. When transferring possession of the samples, the individuals relinquishing and receiving the samples will sign, date, and note the time of transfer on the record. A commercial delivery service (for example, Federal Express) will be identified by company name only. Additionally, the samples will remain in the physical possession of the person assigned to the sample until they are shipped to the laboratory or will be placed in a locked storage facility prior to shipping. The original chain-of-custody record will accompany the sample to the analytical laboratory and will be returned to the Remedial Contractor with the analytical results. A copy of each record will be placed in the project file.

## **7.4 Sample Packaging and Shipping**

Samples will be shipped by overnight courier as environmental samples according to applicable guidance documents and DOT regulations. Sample containers will be prepared according to the U.S. EPA's Specifications and Guidance for Contaminant Free Sample Containers, April 1990. This document is attached to Appendix C to the QAPP.

### **7.4.1 Environmental Samples**

Sample packaging and shipping procedures are described below:

- Secure sample bottle lids with strapping tape or evidence tape. Check that sample label is securely attached.
- Mark volume level on bottle with grease pencil.
- Place bottles in plastic bags.
- Place about 3 inches of inert cushioning material such as vermiculite in bottom of cooler.
- Place containers in cooler in such a way that they do not touch.
- Put VOA vials in Ziploc bag and place them in the center of the cooler.
- Pack plastic Ziploc bags with ice and place in cooler.
- Fill cooler with cushioning material.
- Put paperwork in plastic bags and tape to inside lid of cooler.
- Tape drain shut.
- After acceptance by Federal Express or shipper, wrap cooler completely with strapping tape at two locations. Do not cover any labels.

- Place lab address on top of cooler.
- Put "THIS SIDE UP" labels on all four sides and "FRAGILE" labels on at least two sides. ("FRAGILE" labels are optional for coolers not containing glass bottles.)
- Affix signed custody seals on front right and back left of cooler. Cover seals with wide, clear tape.

#### **7.4.2 Medium or High Concentration Hazardous Waste Samples**

Samples from unclassified drums may require packaging and shipping according to applicable guidance documents and DOT regulations for medium or high concentration hazardous waste samples. Sample packaging and shipping are described below:

- Secure sample jar lids with strapping tape or evidence tape.
- Position jar in Ziploc bag so that tags may be read and seal bag.
- Place 1/2 inch of cushioning material in the bottom of metal can.
- Place jar in can and fill remaining volume of can with cushioning material.
- Close the can using three clips equally spaced to secure the lid.
- Write sample identification number on can lid. Indicate "THIS SIDE UP" by drawing an arrow and place "FLAMMABLE LIQUID N.O.S." label, if appropriate, on can.
- Place 1 inch of packing material in bottom of cooler.
- Place cans in cooler and fill remaining volume of cooler with packing material.
- Put paperwork in plastic bags and tape to inside lid of cooler.

- Tape drain shut.
- After acceptance by the shipper, tape cooler completely around with strapping tape at two locations. Do not cover any labels.
- Place lab address on top of cooler.
- For all medium and high concentration shipments, complete shipper's hazardous material certification form.
- Put "THIS SIDE UP" labels on all four sides, "FLAMMABLE LIQUID N.O.S." or "FLAMMABLE SOLID N.O.S." and "DANGER-PELIGRO" labels on two sides.

Note: "DANGER-PELIGRO" labels should be used only when net quantity of samples in cooler exceed 1 quart (32 ounces) for liquids or 25 pounds for solids.

- Affix custody seals on front right and back left of cooler. Cover seals with wide, clear tape.

**APPENDIX A1**

**INVENTORY SUMMARY TABLES**

**(INVENTORY PERFORMED ON NOVEMBER 13, AND 14, 1992)**

**TABLE 1****TANK INVENTORY SUMMARY TABLE  
PAGE 1 OF 6**

| <b>Tank Number</b> | <b>Height/Length (Ft)</b> | <b>Diameter (Ft)</b> | <b>Thickness (In)</b> | <b>Condition</b> | <b>Contents</b> | <b>Miscellaneous/Comments</b>                                     |
|--------------------|---------------------------|----------------------|-----------------------|------------------|-----------------|---|
| T-1                | 15.35                     | 10.6                 | 3/16                  | Fair             | Clean and dry   | 16 feet of 2-inch piping<br>15 feet of 3-inch piping              |
| T-2                | 18                        | 10                   | 3/16                  | Fair             | Clean and dry   | 15 square feet of insulation<br>5 foot x 5 foot hole cut in side  |
| T-3                | 30                        | 6                    | 1/4                   | Good             | Unknown         | Inaccessible port<br>Riveted steel                                |
| T-4                | 32.2                      | 5.5 avg.             | 1/8                   | Poor             | Clean and dry   | 5,000 gallon tanker<br>Truck-back end cut open<br>Stainless steel |
| T-5                | 33                        | 5.5 avg.             | 3/16                  | Fair             | Empty           | Tanker truck with baffles   |
| T-6                | 31.5                      | 10                   | 3/16                  | Fair             | Unknown         | Inaccessible port   |
| T-7                | 24                        | 8                    | 3/16                  | Poor             | Clean and dry   | Tank has four 6-foot legs   |
| T-8                | 23.5                      | 10.5                 | 1/4                   | Fair             | Unknown         | Inaccessible port<br>Riveted steel                                |
| T-9                | 20                        | 10                   | 1/4                   | Poor             | Unknown         | Inaccessible port<br>Riveted steel                                |

**TABLE 1****TANK INVENTORY SUMMARY TABLE****PAGE 2 OF 6**

| <b>Tank Number</b> | <b>Height/Length (Ft)</b> | <b>Diameter (Ft)</b> | <b>Thickness (In)</b> | <b>Condition</b> | <b>Contents</b>   | <b>Miscellaneous/Comments</b>   |
|--------------------|---------------------------|----------------------|-----------------------|------------------|---|---|
| T-10               | 27                        | 8                    | 3/16                  | Fair             | Clean and dry   |   |
| T-11               | 25.5                      | 4.25                 | 3/16                  | Poor             | Empty with considerable amount of scale                           | 4,000 gallon vacuum tanker truck on wheels<br>Miscellaneous piping and equipment attached |
| T-12               | 24                        | 5.35                 | 3/16                  | Fair             | Empty with minimal scale debris                                   |   |
| T-13               | 22                        | 8                    | 3/16                  | Fair             | Unknown   | Inaccessible port   |
| T-14               | 18                        | 9.5                  | 3/16                  | Poor             | Chemical scale on interior walls<br>1 inch clear liquid on bottom | 5 foot x 3 foot hole cut<br>3 foot x 2 foot hole cut                                      |
| T-15               | 13.5                      | 7.5                  | 3/16                  | Fair             | Clean and dry   |   |
| T-16               | 16                        | 10.4                 | 1/4                   | Fair             | Clean and dry   | Riveted steel   |
| T-17               | 16                        | 13                   | 3/16                  | Fair             | Clean and dry<br>Minimal scale                                    |   |
| T-18               | 12                        | 8                    | 3/16                  | Poor             | Puddled water on bottom;<br>otherwise clean                       |   |
| T-19               | 12                        | 8                    | 3/16                  | Poor             | Clean and dry   |   |
| T-20               | 21                        | 8                    | 3/16                  | Fair             | Unknown   | No visible ports  |



**TABLE 1****TANK INVENTORY SUMMARY TABLE  
PAGE 3 OF 6**

| <b>Tank Number</b> | <b>Height/Length (Ft)</b> | <b>Diameter (Ft)</b> | <b>Thickness (In)</b> | <b>Condition</b> | <b>Contents</b>  | <b>Miscellaneous/Comments</b>   |
|--------------------|---------------------------|----------------------|-----------------------|------------------|--|---|
| T-21               | 35                        | 7                    | 1/4                   | Fair             | Clean and dry  | Riveted steel<br><br>Scale on interior wall<br><br>Note on side of tank painted "PCB Hoses Only"        |
| T-22               | 15.5                      | 10.5                 | 1/8                   | Poor             | Clean and empty<br><br>Minimal scale                     |   |
| T-23               | 21                        | 12.5                 | 3/16                  | Poor             | Clean and dry<br><br>Minimal scale                       |   |
| T-24               | 16                        | 10                   | 3/16                  | Poor             | 1 inch liquid<br><br>Some solid debris<br><br>Tank scale |   |
| T-25               | 15                        | 10.5                 | 3/16                  | Poor             | Clean with minimal solid debris and tank scale           |   |
| T-26               | 32.3                      | 5 avg.               | 1/8                   | Very poor        | Nothing  | Tanker truck with side cut out<br><br>Note on truck: "Licensed Special Waste Hauler - ILL EPA-0295/002" |

**TABLE 1****TANK INVENTORY SUMMARY TABLE****PAGE 4 OF 6**

| <b>Tank Number</b> | <b>Height/Length (Ft)</b> | <b>Diameter (Ft)</b> | <b>Thickness (In)</b> | <b>Condition</b> | <b>Contents</b>  | <b>Miscellaneous/Comments</b>            |
|--------------------|---------------------------|----------------------|-----------------------|------------------|--|--|
| T-27               | 12                        | 8                    | 3/16                  | Poor             | Empty except for roof debris on bottom<br><br>1 to 2 inches of liquid on bottom;<br>most likely rain water | Roof is missing (rusted away)            |
| T-28               | 25.5                      | 9                    | 1/4                   | Fair             | Empty except for solid debris and tank scale   | Riveted steel                            |
| T-29               | 30                        | 10.5                 | 3/16                  | Fair             | Unknown  | Inaccessible port                        |
| T-30               | 20.3                      | 10                   | 1/4                   | Fair             | Unknown  | Riveted steel<br><br>Inaccessible port   |
| T-31               | 24.5                      | 10.5                 | 3/16                  | Poor             | 1 inch liquid on bottom and minimal scale  |  |
| T-32               | 16                        | 8                    | 1/4                   | Poor             | Unknown  | Inaccessible port<br><br>Severely dented |
| T-33               | 27                        | 8                    | 3/16                  | Fair             | Clean and dry with minimal tank scale  | Painted on side "Caution PCBs"           |
| T-34               | 16                        | 13                   | 3/16                  | Poor             | Clean and empty with minimal scale   | Miscellaneous piping along side          |
| T-35               | 6.25                      | 5                    | 3/16                  | Fair             | 1/2 inch liquid with tank scale and crust  |  |
| T-36               | 19                        | 6                    | 3/16                  | Fair             | Clean and dry  | Built 1971                               |

**TABLE 1****TANK INVENTORY SUMMARY TABLE****PAGE 5 OF 6**

| <b>Tank Number</b> | <b>Height/Length (Ft)</b> | <b>Diameter (Ft)</b> | <b>Thickness (In)</b> | <b>Condition</b> | <b>Contents</b>                           | <b>Miscellaneous/Comments</b>                          |
|--------------------|---------------------------|----------------------|-----------------------|------------------|---|--|
| T-37               | 12                        | 5.5                  | 3/16                  | Fair             | Clean and dry                             | 8 feet of pipe along tank                              |
| T-38               | 12                        | 5.5                  | 3/16                  | Fair             | Unknown                                   | Inaccessible port                                      |
| T-39               | 13                        | 9.5                  | 3/16                  | Fair             | 2 inch tank scale<br>Solid debris unknown |  |
| T-40               | 12                        | 5.5                  | 3/16                  | Fair             | Unknown                                   | Inaccessible port                                      |
| T-41               | 13                        | 9.5                  | 3/16                  | Fair             | Clean with minimal scale                  |  |
| T-42               | 13                        | 9.5                  | 3/16                  | Fair             | Clean and empty                           |  |
| T-43               | 13                        | 9.5                  | 3/16                  | Fair             | Clean and empty                           |  |
| T-44               | 6                         | 5.5                  | 3/16                  | Fair             | Clean and dry<br>Minimal scale            |  |
| T-45               | 12.2                      | 3.8                  | 3/16                  | Fair             | Unknown                                   | Inaccessible port                                      |
| T-46               | 6                         | 6                    | 3/16                  | Poor             | Clean and dry                             | Wrapped in foam insulation with miscellaneous piping   |
| T-47               | 6                         | 4.5                  | 3/16                  | Poor             | Clean and dry with minimal tank scale     | Wrapped in foam insulation with miscellaneous piping   |
| T-48               | 11.5                      | 5                    | 1/4                   | Fair/Good        | 1/4 inch liquid; otherwise clean          | Stainless steel construction with miscellaneous piping |
| T-49               | 6                         | 4                    | 3/16                  | Fair             | Clean and dry                             | Miscellaneous piping                                   |

**TABLE 1****TANK INVENTORY SUMMARY TABLE  
PAGE 6 OF 6**

| <b>Tank Number</b> | <b>Height/Length (Ft)</b> | <b>Diameter (Ft)</b> | <b>Thickness (In)</b> | <b>Condition</b> | <b>Contents</b> | <b>Miscellaneous/Comments</b>       |
|--------------------|---------------------------|----------------------|-----------------------|------------------|-----------------|-------------------------------------|
| T-50               | 6                         | 6                    | 3/16                  | Fair             | Clean and dry   | Wrapped in foam insulation          |
| T-51               | 6                         | 4.5                  | 3/16                  | Fair             | Clean and dry   | Wrapped in foam insulation          |
| T-52               | 30                        | 6                    | 3/8                   | Fair             | Unknown         | Riveted steel<br>Inaccessible ports |
| T-53               | 22                        | 7.5                  | 3/16                  | Fair             | Unknown         | Inaccessible ports                  |

**Notes**

1. All tanks and piping are constructed of carbon steel unless otherwise noted.
2. All tanks had no detectable PID or LEL/O<sub>2</sub> indications other than background readings.
3. Considerable amount of brush exists between/around tanks including trees up to 4 inches in diameter.
4. A concrete and steel tank stand, forklift, and other various steel debris is scattered about the tank area.
5. References to measurements (height, diameter, and thickness of tank) are approximate.

| TABLE 2                                   |                   |                                      |  |
|---|-------------------|--------------------------------------|--|
| DRUM STORAGE AREA INVENTORY SUMMARY TABLE |                   |                                      |  |
| Drum Storage Area                         | Quantity of Drums | Condition                            | Comments   |
| 1   | 240 ±             | Poor:<br><br>Deteriorated            | Drums from the Enviro-Chem Site, the Northside Sanitary Landfill, and the Third Site contained soil cuttings from drilling operations, groundwater, decontamination water, and chemical protective clothing. Several drums are unmarked as to their contents or source of contents. Some drums have rusted open and now contain nothing. |
| 2   | 10                | New:<br><br>Able to be shipped as is | Eight drums contain soil cuttings, decontamination water, groundwater, and chemical protective clothing from activities on the Enviro-Chem Site generated by AWD. Two unused drums remain empty.   |

#### Notes

1. All drums are 55-gallon.
2. Approximately 20 other drums are located in various other areas onsite.

**TABLE 3****STRUCTURE INVENTORY SUMMARY TABLE  
PAGE 1 OF 3**

| <b>Building</b>                   | <b>Dimensions<br/>(Ft)</b> | <b>Building Materials</b>  | <b>Contents</b>  |
|-----------------------------------|----------------------------|--|--|
| <b>A-Frame House</b>              | <b>28 x 20 x 18 H</b>      | <b>All wood construction with asphalt shingles; above ground construction; no foundation</b> |  |
| <b>Lower Floor;<br/>West Room</b> | <b>12 x 18</b>             |  | <b>Ten 50-lb bags of grass fertilizer<br/>Eight 50-lb bags of plant food<br/>Three gallons of pesticide<br/>Two gallons of paint<br/>One 55-gallon drum; unknown contents<br/>One tire<br/>Six milk crates<br/>One 5-foot book shelf<br/>Ten square feet of rubber matting<br/>Several florescent light fixtures (4-foot long)<br/>Three boxes of florescent light tubes (4-foot long)<br/>Several yard hand tools<br/>Other miscellaneous debris</b>  |
| <b>Lower Floor;<br/>East Room</b> | <b>12 x 18</b>             |  | <b>5 foot x 3 foot kitchen cabinet unit<br/>One kitchen sink<br/>One table band saw<br/>One wall air conditioning unit<br/>Two work tables<br/>Three chairs<br/>Two lawn fertilizer spreaders<br/>100 feet of 1-inch PVC tubing<br/>Several boxes of sorbent pads (24 inch x 24 inch) and<br/>8-inch diameter x 6 feet long sorbent sock<br/>One tire<br/>One 55-gallon tub<br/>Two rolls of carpet pad (6 foot x 20-inch diameter)<br/>Miscellaneous 5-gallon buckets of debris<br/>Loose fertilizer on floor</b> |

**TABLE 3****STRUCTURE INVENTORY SUMMARY TABLE  
PAGE 2 OF 3**

| <b>Building</b>                  | <b>Dimensions<br/>(Ft)</b> | <b>Building Materials</b>  | <b>Contents</b>   |
|----------------------------------|----------------------------|--|---|
| <b>Upper Level;<br/>One Room</b> | <b>24 x 10</b>             | <b>9 inch x 9 inch vinyl floor tile</b>  | <b>Three boxes of sorbent pads (24 inch x 24 inch)<br/>Miscellaneous debris (basically clean and empty)</b>   |
| <b>Outside; West</b>             |                            |  | <b>One office desk<br/>One fertilizer spreader<br/>Wood debris<br/>Miscellaneous debris</b>   |
| <b>Outside; East</b>             |                            |  | <b>Two air conditioner units<br/>One office desk<br/>Miscellaneous debris</b>   |
| <b>Process Building</b>          | <b>76 x 36/30 x 32 H</b>   |  |   |
| <b>Room 1</b>                    | <b>30 x 18 x 16 H</b>      | <b>One cinder block wall (16 feet high x 30 feet)<br/>Eight 8 foot x 8 inch steel beams<br/>150 feet of 6-inch channel steel<br/>Aluminum sheeting on walls and roof with fiberglass insulation<br/>Concrete floor/foundation</b>  | <b>One boiler (16 foot x 6 1/2 foot diameter on 8-inch steel I-beam frame)<br/>One 5 foot x 3 foot fuel tank<br/>One 8 foot x 4 foot electrical panel</b>           |
| <b>Room 2</b>                    | <b>30 x 27 x 16 H</b>      | <b>Two cinder block walls (one between Rooms 1 and 2 accounted for in Room 1 listing (16 feet high x 30 feet)<br/>Aluminum walls on east and west sides<br/>Eight 8 inch x 30 foot steel beams<br/>Two 8 inch to 18 inch x 30 foot main beams<br/>Four 8 inch x 12 foot steel upright beams<br/>120 feet of 6-inch steel channel beams<br/>Concrete floor/foundation</b> | <b>Various steel piping<br/>Three 10 foot x 8 foot book shelves (2 steel/1 wooden)<br/>One snowmobile<br/>Fifteen 4 foot x 8 foot styrofoam sheeting insulation</b> |

**TABLE 3****STRUCTURE INVENTORY SUMMARY TABLE  
PAGE 3 OF 3**

| <b>Building</b> | <b>Dimensions<br/>(Ft)</b> | <b>Building Materials</b>  | <b>Contents</b>  |
|-----------------|----------------------------|--|--|
| <b>Room 3</b>   | <b>36 x 33 x 32 H</b>      | <b>Two cinder block walls (between Rooms 2 and 3<br/>accounted for in Room 2); the other wall is 23 feet<br/>high x 36 feet<br/>Aluminum walls on east and west sides<br/>Partially missing aluminum roof<br/>Wooden roof supports<br/>Concrete floor/foundation</b> | <b>One 6-foot exhaust fan built in ceiling<br/>Forty florescent light fixtures (4-foot long)<br/>Twelve steel bookshelves<br/>Six tires<br/>Forty 6-inch PVC elbows and tees<br/>Rolls of fiberglass insulation<br/>Various other debris</b> |

**Note**

1. All concrete floors/foundations will be left intact.
2. There was 2 to 6 inches of water present on the floor of Room 1 of the process building during this inventory. However, the amount of water will fluctuate based on weather conditions.
3. There is a power pole (with two transformers) located outside the northwest corner of Room 1 of the process building.



**TABLE 4****MISCELLANEOUS DEBRIS AREA INVENTORY SUMMARY TABLE  
(SEE DRAWINGS FOR LOCATIONS)  
PAGE 1 OF 2**

| Miscellaneous Debris Area | Debris Item  |
|---------------------------|--|
| 1                         | Seven 55-gallon drums - unknown contents<br>One 4 foot x 4 foot utility sink<br>Pile of cardboard<br>Pile of pieces of wood<br>Painting tools  |
| 2                         | Ten 12-foot wood planks<br>One 18 foot x 10 inch steel lifting beam<br>Twelve 10 foot x 3 foot aluminum sheets   |
| 3                         | Scaffolding material - planks, stands, ladders<br>One riding lawn mower<br>One 30-gallon fuel tank<br>Fifteen feet of 5-inch steel pipe<br>Four 10 foot x 3 foot aluminum sheets<br>One metal storage box (4 foot x 3 foot x 2 foot)<br>Two rolls of chicken wire (2 1/2 foot x 18 inches)<br>One roll of cyclone fence (4 foot x 20 inches)<br>Two 20-foot aluminum gutters<br>Six prefab roof supports (25 foot x 4 foot)  |
| 4                         | Six tires<br>One lawn mower<br>Four wooden planks<br>One snowmobile carcass  |
| 5                         | 10 foot x 10 foot x 2 inch aluminum roof panel<br>Eight 55-gallon drums - contents unknown<br>Wood pile 20 square feet x 4 inches high<br>600 feet of 1-inch PVC piping<br>300 feet of 6-inch PVC piping<br>30 feet of 8-inch PVC piping<br>100 feet of 2-inch galvanized steel pipe<br>200 feet of 4-inch corrugated flexline pipe<br>2-inch steel tubing/framework (100 feet total length)<br>One air compressor<br>Seventeen 1-foot sections of terracotta pipe<br>300 feet of 1-inch PVC well tubing<br>One 30-gallon tank<br>Twelve concrete parking blocks (6 feet long) |

**TABLE 4**

**MISCELLANEOUS DEBRIS AREA INVENTORY SUMMARY TABLE  
(SEE DRAWINGS FOR LOCATIONS)  
PAGE 2 OF 2**

| Miscellaneous Debris Area | Debris Item   |
|---------------------------|---|
| 6                         | Three 3 foot x 15 foot sheets of aluminum<br>One 6 foot x 3 foot book shelf<br>One 10 foot x 12 foot aluminum wall<br>One snowmobile carcass<br>Scattered insulation<br>One diesel truck engine<br>Two truck tires<br>One aluminum box 10 foot x 8 foot x 8 foot (storage shed)<br>10 foot x 12 foot area of machinery parts<br>One 55-gallon drum - contents unknown |

| TABLE 5  |  |  |
|--|--|--|
| SOIL VAPOR EXTRACTION PILOT STUDY AREA INVENTORY SUMMARY TABLE |  |  |
| Item   | Quantity   | Debris in Area   |
| Pilot Vapor Extraction System                                  | 100 feet of 4-inch exposed PVC pipe<br>80 feet of 4-inch buried PVC pipe | 8 railroad timbers<br>20 tires<br>30 feet of 4-inch corrugated flex line |

**Note**

1. Buried pipe not included in this contract.

**TABLE 6****OTHER SITE DEBRIS INVENTORY SUMMARY TABLE**

| <b>Item</b>                                | <b>Approximate Quantity</b>                | <b>Location</b>               |
|--|--|-------------------------------|
| <b>Dismantled modular tanks</b>            | 450 square feet aluminum and plastic liner | Southern concrete pad         |
| <b>Wood pile</b>                           | 20 feet x 10 feet x 4 feet high            | Southern concrete pad         |
| <b>Various pieces of aluminum sheeting</b> | 20   | Entire site                   |
| <b>Bentonite</b>                           | 1 pallet (500 lbs)                         | Northwest of Process Building |

**APPENDIX B**

**TOXICITY CHARACTERISTIC  
LEACHING PROCEDURE (TCLP)**

## **Toxicity Characteristic Leaching Procedure (TCLP)**

When the sample contains no filterable liquid, the TCLP method is performed as follows:

1. Obtain a representative 100 gram sample of solid material.
2. Crush material to  $<9.5$  mm, if necessary, and place in extraction vessel.
3. Determine appropriate extraction medium:
  - a. Weigh out 5 grams subsample of sample; reduce particle size to  $<1$  mm, if required; place sample in a 500 mL beaker.
  - b. Add 96.5 mL of distilled/deionized water (ASTM Type II).
  - c. Stir sample vigorously for 5 minutes with magnetic stirrer.
  - d. Measure pH, and, if pH is  $\leq 5$ , use Extraction Fluid No. 1.
  - e. If pH  $> 5$ , add 3.5 mL 1.0 N HCl; slurry for 30 seconds; heat to  $50^{\circ}\text{C}$  for 10 minutes.
  - f. Allow mixture to cool to room temperature and measure pH.
  - g. if pH  $\leq 5$ , use Extraction Fluid No. 1. and if pH  $> 5$ , use Extraction Fluid No. 2.
4. Add amount of extraction fluid selected in Step 3 equal to 20 times the weight of the solid residue.
5. Close extraction vessel, and agitate in rotary extractor device at  $30 \pm 2$  rpm for 18 hours, maintaining the temperature at  $22 \pm 3^{\circ}\text{C}$ .

6. Filter material through a 0.6 to 0.8  $\mu\text{m}$  glass fiber filter.
7. Analyze or preserve filtrate as required.

If the residue sample contains filterable liquid, the sample is first separated into its component phases, and the above procedure is carried out on the solid phase. Then if the initial filtrate and solid extract are compatible (i.e., will not form multiple phases or precipitates on combination), they are analyzed separately, and the results are mathematically combined to yield the total leachable composition.

Since the pH of the waste determines the nature of the extraction fluid used, either Extraction Fluid No. 1 or No. 2, it is important to define the TCLP definition of these fluids:

- Extraction Fluid No. 1 is made by combining 64.3 mL of 1.0 N NaOH and 5.7 mL glacial acetic acid to the appropriate volume of water and diluting to a volume of 1 liter. The pH of this fluid should be 4.93  $\pm$  0.02.
- Extraction Fluid No. 2 is made by diluting 5.7 mL glacial acetic acid with ASTM Type 2 water to a volume of 1 liter. The pH of this fluid should be 2.88  $\pm$  0.02.

**APPENDIX C**  
**CONTAMINANT-FREE SAMPLE CONTAINERS**



**SPECIFICATIONS  
AND  
GUIDANCE  
FOR  
CONTAMINANT-FREE SAMPLE CONTAINERS**

**APRIL 1992**

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## SECTION I

### INTRODUCTION

In August 1989, the Environmental Protection Agency's (EPA) Office of Emergency and Remedial Response (OERR) decentralized Superfund's Sample Container Repository program (OSWER Directive #9240.0-05). In conjunction with the decentralization of Superfund's bottle program, OERR issued specifications and guidance for preparing contaminant-free sample containers to assist the Regions in obtaining appropriate sample containers from commercially available suppliers.

The April 1992 version of "Specifications and Guidance for Contaminant-Free Sample Containers" revises the specifications and provides a single source of standardized specifications and guidance on appropriate cleaning procedures for preparing contaminant-free sample containers that meet all Contract Laboratory Program (CLP) detection/quantitation limits, including those for low concentration analyses.<sup>1</sup> Although the specifications and guidance procedures contained in this document are based on CLP low concentration requirements, they also are suitable for use in other analytical programs.

Specifications and guidance for preparing contaminant-free sample containers are provided in the sections that follow and are intended to describe one approach for obtaining cleaned, contaminant-free sample containers for use by groups performing sample collection activities under Superfund and other hazardous waste programs. Although other cleaning procedures may be used, sample containers must meet the criteria specified in Section II. In certain instances, the user of the sample containers may require exact adherence to the cleaning procedures and/or quality control analysis described in this document. In other instances, the user may require additional or different cleaning procedures and/or quality control analysis of the sample containers. The specific needs of the bottle user will determine the requirements for the cleaning and quality control analysis of the sample containers as long as the minimum criteria are met. It is the responsibility of the bottle user to define the sample container preparation, cleaning, and quality control requirements.

The document has been extensively reviewed and revised since the August 1989 iteration, and important enhancements have been incorporated, including:

- Removing references to the color of the closures;
- Allowing the use of polypropylene closures as an alternative to

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<sup>1</sup> Because this document does not address the procurement of contaminant-free sample containers, the title was changed from "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers" to "Specifications and Guidance for Contaminant-Free Sample Containers."

phenolic closures;

- Referencing CLP Low Concentration Organics and Inorganics Statements of Work for the analysis of calibration verification solutions and blanks;
- Including cleaning and quality control procedures for fluoride and nitrate/nitrite;
- Removing the hexane rinse from the cleaning procedure for container types A, E, F, G, H, J, and K (semivolatile organics, pesticides, metals, cyanide, and fluoride in soils and water);
- Adding the recommendation that the bottle vendor establish and submit a Quality Assurance Plan (QAP);
- Changing the QA/QC documentation requirements so that copies of the raw data from the analyses of the QC containers are available upon request and not automatically sent to the bottle purchaser;
- Changing the permanent lot number assignment to a nine digit number from an eight digit number, where the extra digit represents the analysis parameter;
- Adding Chemical Abstract Services (CAS) registry number for the inorganic analytes in Table 1; and
- Recommending an annual demonstration of the bottle vendor's ability to meet detection limits and establish reproducibility of the cleaning techniques.

OEER and the EPA Regions decided to use the most stringent CLP requirements available to set the specifications for obtaining contaminant-free sample containers. As a result, the CLP Inorganics and Organics Low Concentration Statement of Work (SOW) requirements were selected as the basis for these specifications. Major factors in this decision included the desire to have a set of bottle cleaning specifications that met or exceeded all analytical requirements and the related need to avoid potential misuse of cleaned bottles (e.g., using a container cleaned by a multi-concentration procedure for a low concentration sample). OEER will reevaluate this decision if the low concentration requirements are deemed to be too stringent.

Most environmental sampling and analytical applications offer numerous opportunities for sample contamination. For this reason, contamination is a common source of error in environmental measurements. The sample container itself represents one such source of sample contamination. Hence, it is vital that sample containers used within the Superfund program meet strict specifications established to minimize contamination which could affect subsequent analytical determinations. Superfund sampling and analysis activities require all component materials (caps, liners, septa, packaging materials, etc.) provided by the bottle preparer to meet the criteria limits of the bottle specifications listed within Section II.

Section III provides guidance on cleaning procedures for preparing contaminant-free sample containers that meet the specifications contained in Section II. The procedures provided in this section are intended to provide sample containers that meet all current CLP Low Concentration Inorganics and Organics detection/quantitation levels.

In selecting cleaning procedures for sample containers, it is important to consider all of the parameters of interest. Although a given cleaning procedure may be effective for one parameter or type of analysis, it may be ineffective for another. When multiple determinations are performed on a single sample or on a subsample from a single container, a cleaning procedure may actually be a source of contamination for some analytes while minimizing contamination in others. It should be the responsibility of the bottle supplier to verify that the cleaning procedures actually used satisfy the quality control requirements set forth in Section IV.

Two aspects of quality assurance (i.e., quality control and quality assessment) must be applied to sample containers as well as to the analytical measurements. Quality control includes the application of good laboratory practices and standard operating procedures especially designed for the cleaning of sample containers. The cleaning operation should be based on protocols especially designed for specific contaminant problems. Strict adherence to these cleaning protocols is imperative. Quality assessment of the cleaning process depends largely on monitoring for adherence to the respective protocols. Because of their critical role in the quality assessment of the cleaning operation, protocols must be carefully designed and followed. Guidance is provided in Section IV on design and implementation of quality assurance and quality control protocols.

## SECTION II

### SAMPLE CONTAINER AND COMPONENT MATERIAL SPECIFICATIONS

This Section identifies sample containers commonly used in the Superfund program and provides specifications for contaminant-free sample containers for each bottle type.

#### A. CONTAINER MATERIAL

A variety of factors affect the choice of containers and cap material. These include resistance to breakage, size, weight, interferences with analytes of interest, cost, and availability.

Container types A through L (Figure 1, pages 7-8) are designated as the type of sample containers that have been used successfully in the past. Kimax or Pyrex brand borosilicate glass is inert to most materials and is recommended where glass containers are used (i.e., pesticides and other organics). Conventional polyethylene is recommended when plastic is acceptable because of its lower cost and lower adsorption of metal ions. The specific sampling situation will determine the use of plastic or glass.

While the sample containers shown in Figure 1 are utilized primarily for Superfund sampling activities, they also may be used for sampling activities under other programs, such as the Resource Conservation and Recovery Act (RCRA).

#### B. MAXIMUM CONTAMINANT LEVEL SPECIFICATIONS FOR SAMPLE CONTAINERS

The CLP, through a series of technical caucuses, has established Inorganic Contract Required Detection Limits (CRDL) and organic Contract Required Quantitation Limits (CRQL) which represent the minimum quantities needed to support the hazardous substance identification and monitoring requirements necessary for remedial and other actions at hazardous waste sites.

For inorganic sample containers, the CRDLs listed in Table 1, page 9, are the specifications for maximum trace metal contamination. Concentration at or above these limits on any parameter should preclude these containers from use in collecting inorganic samples.

The CRQL specifications for organic sample containers are listed in Table 2, pages 10-14. When the CRQL in Table 2 is multiplied by the appropriate factor listed below, the resulting value then represents the maximum concentration allowed for particular sample containers based on organic CLP sample sizes for routine analyses.

| <u>Container Type</u> | <u>Multiple of CRQL</u> |
|-----------------------|-------------------------|
| A                     | 1.0                     |
| B                     | 0.5                     |
| D                     | 10.0                    |
| E                     | 8.0                     |
| F                     | 4.0                     |
| G                     | 2.0                     |
| H                     | 0.5                     |
| J                     | 0.5                     |
| K                     | 2.0                     |

The philosophy used for determining the maximum permissible amount of contamination in a sample container was to consider the number of aliquots of sample that are available in the container and assume that the contamination present would be uniformly distributed in all of the aliquots. This assumption, and the assumption that there should be no more than one-half the CRQL contributed by the container, resulted in the establishment of contamination limits by container type. For example, the volume of container type D is sufficient to allow 20 volatile determinations. Therefore, if 10 times the CRQL of contaminant is present in the cleaned bottle, each aliquot tested will contain one-half of the CRQL of contaminant due to the contribution from the bottle.

#### C. GROSS CONTAMINATION

Gross contamination is defined as greater than two hundred times the acceptable concentration values in Tables 1 or 2 (multiplied by the appropriate factor), unless the cleaning procedure is successful in reducing the amount of contamination to within specifications. If this is not achieved, the grossly contaminated materials should be discarded and replaced to prevent cross contamination with other batches of containers. The bottle preparer should inspect all materials to ensure conformance with the required specifications.

FIGURE 1

SAMPLE CONTAINER  
SPECIFICATIONS

Container

Type Specifications

---

- A     Container: 80-oz amber glass, ring handle bottle/jug, 38-mm neck finish.  
      Closure: polypropylene or phenolic cap, 38-430 size; 0.015-in Teflon liner.  
      Total Weight: 2.45 lbs.
- B     Container: 40-mL glass vial, 24-mm neck finish.  
      Closure: polypropylene or phenolic, open-top, screw cap, 15-cm opening, 24-400 size.  
      Septum: 24-mm disc of 0.005-in Teflon bonded to 0.120-in silicon for total thickness of 0.125-in.  
      Total Weight: 0.72 oz.
- C     Container: 1-L high-density polyethylene, cylinder-round bottle, 28-mm neck finish.  
      Closure: polyethylene cap, ribbed, 28-410 size; F217 polyethylene liner.  
      Total Weight: 1.89 oz.
- D     Container: 120-mL wide mouth, glass vial, 48-mm neck finish.  
      Closure: polypropylene cap, 48-400 size; 0.015-in Teflon liner.  
      Total Weight: 4.41 oz.
- E     Container: 16-oz tall, wide mouth, straight-sided, flint glass jar, 63-mm neck finish.  
      Closure: polypropylene or phenolic cap, 63-400 size; 0.015-in Teflon liner.  
      Total Weight: 9.95 oz.
- F     Container: 8-oz short, wide mouth, straight-sided, flint glass jar, 70-mm neck finish.  
      Closure: polypropylene or phenolic cap, 70-400 size; 0.015-in Teflon liner.  
      Total Weight: 7.55 oz.



FIGURE 1

SAMPLE CONTAINER  
SPECIFICATIONS  
(Continued)

| Container<br>Type | Specifications   |
|-------------------|--|
| G                 | <p><u>Container:</u> 4-oz tall, wide mouth,<br/>straight-sided, flint glass jar,<br/>48-mm neck finish.</p> <p><u>Closure:</u> polypropylene or phenolic cap,<br/>48-400 size; 0.015-in Teflon liner.</p> <p><u>Total Weight:</u> 4.70 oz.</p>   |
| H                 | <p><u>Container:</u> 1-L amber, Boston round, glass<br/>bottle, 33-mm pour-out neck finish.</p> <p><u>Closure:</u> polypropylene or phenolic cap,<br/>33-430 size; 0.015-in Teflon liner.</p> <p><u>Total Weight:</u> 1.11 lbs.</p>              |
| J                 | <p><u>Container:</u> 32-oz tall, wide mouth,<br/>straight-sided, flint glass jar,<br/>89-mm neck finish.</p> <p><u>Closure:</u> polypropylene or phenolic cap,<br/>89-400 size; 0.015-in Teflon liner.</p> <p><u>Total Weight:</u> 1.06 lbs.</p> |
| K                 | <p><u>Container:</u> 4-L amber glass, ring handle<br/>bottle/jug, 38-mm neck finish.</p> <p><u>Closure:</u> polypropylene or phenolic cap,<br/>38-430 size; 0.015-in Teflon liner.</p> <p><u>Total Weight:</u> 2.88 lbs.</p>                     |
| L                 | <p><u>Container:</u> 500-mL high-density polyethylene,<br/>cylinder-round bottle, 28-mm neck finish.</p> <p><u>Closure:</u> polypropylene cap, ribbed, 28-410 size;<br/>F217 polyethylene liner.</p> <p><u>Total Weight:</u> 1.20 oz.</p>        |

**TABLE 1**  
**INORGANIC ANALYTE**  
**SPECIFICATIONS**

| Analyte             | CAS Number | Contract Required<br>Detection Limits <sup>1</sup><br>(µg/L) |
|---------------------|------------|--|
| 1. Aluminum         | 7429-90-5  | 100  |
| 2. Antimony         | 7440-36-0  | 5  |
| 3. Arsenic          | 7440-38-2  | 2  |
| 4. Barium           | 7440-39-3  | 20   |
| 5. Beryllium        | 7440-41-7  | 1  |
| 6. Cadmium          | 7440-43-9  | 1  |
| 7. Calcium          | 7440-70-2  | 500  |
| 8. Chromium         | 7440-47-3  | 10   |
| 9. Cobalt           | 7440-48-4  | 10   |
| 10. Copper          | 7440-50-8  | 10   |
| 11. Iron            | 7439-89-6  | 500  |
| 12. Lead            | 7439-92-1  | 2  |
| 13. Magnesium       | 7439-95-4  | 500  |
| 14. Manganese       | 7439-96-5  | 10   |
| 15. Mercury         | 7439-97-6  | 0.2  |
| 16. Nickel          | 7440-02-0  | 20   |
| 17. Potassium       | 7440-09-7  | 750  |
| 18. Selenium        | 7782-49-2  | 3  |
| 19. Silver          | 7440-22-4  | 10   |
| 20. Sodium          | 7440-23-5  | 500  |
| 21. Thallium        | 7440-28-0  | 10   |
| 22. Vanadium        | 7440-62-2  | 10   |
| 23. Zinc            | 7440-66-6  | 20   |
| 24. Cyanide         | 57-12-5    | 10   |
| 25. Fluoride        | 16984-48-8 | 200  |
| 26. Nitrate/Nitrite | 1-00-5     | 100  |

<sup>1</sup>CRDLs are based on the CLP Inorganics Low Concentration SOW

**TABLE 2**  
**ORGANIC COMPOUND**  
**SPECIFICATIONS**

| Volatiles |                           | CAS Number | Contract Required<br>Quantitation Limits <sup>1</sup><br>(µg/L) |
|-----------|---------------------------|------------|---|
| 1.        | Chloromethane             | 74-87-3    | 1   |
| 2.        | Bromomethane              | 74-83-9    | 1   |
| 3.        | Vinyl Chloride            | 75-01-4    | 1   |
| 4.        | Chloroethane              | 75-00-3    | 1   |
| 5.        | Methylene Chloride        | 75-09-2    | 2   |
| 6.        | Acetone                   | 67-64-1    | 5   |
| 7.        | Carbon Disulfide          | 75-15-0    | 1   |
| 8.        | 1,1-Dichloroethane        | 75-35-4    | 1   |
| 9.        | 1,1-Dichloroethane        | 75-34-3    | 1   |
| 10.       | cis-1,2-Dichloroethane    | 156-59-4   | 1   |
| 11.       | trans-1,2-Dichloroethane  | 156-60-5   | 1   |
| 12.       | Chloroform                | 67-66-3    | 1   |
| 13.       | 1,2-Dichloroethane        | 107-06-2   | 1   |
| 14.       | 2-Butanone                | 78-93-3    | 5   |
| 15.       | Bromochloromethane        | 74-97-5    | 1   |
| 16.       | 1,1,1-Trichloroethane     | 71-55-6    | 1   |
| 17.       | Carbon Tetrachloride      | 56-23-5    | 1   |
| 18.       | Bromodichloromethane      | 75-27-4    | 1   |
| 19.       | 1,2-Dichloropropane       | 78-87-5    | 1   |
| 20.       | cis-1,3-Dichloropropene   | 10061-01-5 | 1   |
| 21.       | Trichloroethene           | 79-01-6    | 1   |
| 22.       | Dibromochloromethane      | 124-48-1   | 1   |
| 23.       | 1,1,2-Trichloroethane     | 79-00-5    | 1   |
| 24.       | Benzene                   | 71-43-2    | 1   |
| 25.       | trans-1,3-Dichloropropene | 10061-02-6 | 1   |
| 26.       | Bromoform                 | 75-25-2    | 1   |
| 27.       | 4-Methyl-2-pentanone      | 108-10-1   | 5   |
| 28.       | 2-Hexanone                | 591-78-6   | 5   |
| 29.       | Tetrachloroethene         | 127-18-4   | 1   |
| 30.       | 1,1,2,2-Tetrachloroethane | 79-34-5    | 1   |

<sup>1</sup>CRQLs are based on the CLP Organics Low Concentration SOW

TABLE 2  
ORGANIC COMPOUND  
SPECIFICATIONS  
(Continued)

| Volatiles |                             | CAS Number | Contract Required<br>Quantitation Limits <sup>1</sup><br>(µg/L) |
|-----------|-----------------------------|------------|---|
| 31.       | 1,2-Dibromoethane           | 106-93-4   | 1   |
| 32.       | Toluene                     | 108-88-3   | 1   |
| 33.       | Chlorobenzene               | 108-90-7   | 1   |
| 34.       | Ethylbenzene                | 100-41-4   | 1   |
| 35.       | Styrene                     | 100-42-5   | 1   |
| 36.       | Xylenes (total)             | 1330-20-7  | 1   |
| 37.       | 1,3-Dichlorobenzene         | 541-73-1   | 1   |
| 38.       | 1,4-Dichlorobenzene         | 106-46-7   | 1   |
| 39.       | 1,2-Dichlorobenzene         | 95-50-1    | 1   |
| 40.       | 1,2-Dibromo-3-chloropropane | 96-12-8    | 1   |

<sup>1</sup>CRQLs are based on the CLP Organics Low Concentration SOW

TABLE 2  
ORGANIC COMPOUND  
SPECIFICATIONS  
(Continued)

| Semivolatiles |                               | CAS Number | Contract Required<br>Quantitation Limits <sup>1</sup><br>(µg/L) |
|---------------|-------------------------------|------------|---|
| 1.            | Phenol                        | 108-95-2   | 5   |
| 2.            | bis-(2-Chlorethyl) ether      | 111-44-4   | 5   |
| 3.            | 2-Chlorophenol                | 95-57-8    | 5   |
| 4.            | 2-Methylphenol                | 95-48-7    | 5   |
| 5.            | 2,2'-oxybis-(1-Chloropropane) | 108-60-1   | 5   |
| 6.            | 4-Methylphenol                | 106-44-5   | 5   |
| 7.            | N-Nitroso-di-n-dipropylamine  | 621-64-7   | 5   |
| 8.            | Hexachloroethane              | 67-72-1    | 5   |
| 9.            | Nitrobenzene                  | 98-95-3    | 5   |
| 10.           | Isophorone                    | 78-59-1    | 5   |
| 11.           | 2-Nitrophenol                 | 88-75-5    | 5   |
| 12.           | 2,4-Dimethylphenol            | 105-67-9   | 5   |
| 13.           | bis-(2-Chloroethoxy)methane   | 111-91-1   | 5   |
| 14.           | 2,4-Dichlorophenol            | 120-83-2   | 5   |
| 15.           | 1,2,4-Trichlorobenzene        | 120-82-1   | 5   |
| 16.           | Naphthalene                   | 91-20-3    | 5   |
| 17.           | 4-Chloroaniline               | 106-47-8   | 5   |
| 18.           | Hexachlorobutadiene           | 87-68-3    | 5   |
| 19.           | 4-Chloro-3-methylphenol       | 59-50-7    | 5   |
| 20.           | 2-Methylnaphthalene           | 91-57-6    | 5   |
| 21.           | Hexachlorocyclopentadiene     | 77-47-4    | 5   |
| 22.           | 2,4,6-Trichlorophenol         | 88-06-2    | 5   |
| 23.           | 2,4,5-Trichlorophenol         | 95-95-4    | 20  |
| 24.           | 2-Chloronaphthalene           | 91-58-7    | 5   |
| 25.           | 2-Nitroaniline                | 90 73-4    | 20  |
| 26.           | Dimethylphthalate             | 131-11-3   | 5   |
| 27.           | Acenaphthylene                | 208-96-8   | 5   |
| 28.           | 2,6-Dinitrotoluene            | 606-20-2   | 5   |
| 29.           | 3-Nitroaniline                | 99-09-2    | 20  |
| 30.           | Acenaphthene                  | 83-32-9    | 5   |

<sup>1</sup>CRQLs are based on the CLP Organics Low Concentration SOW

TABLE 2  
ORGANIC COMPOUND  
SPECIFICATIONS  
(Continued)

| Semivolatiles |                             | CAS Number | Contract Required<br>Quantitation Limits <sup>1</sup><br>(µg/L) |
|---------------|-----------------------------|------------|---|
| 31.           | 2,4-Dinitrophenol           | 51-28-5    | 20  |
| 32.           | 4-Nitrophenol               | 100-02-7   | 20  |
| 33.           | Dibenzofuran                | 132-64-9   | 5   |
| 34.           | 2,4-Dinitrotoluene          | 121-14-2   | 5   |
| 35.           | Diethylphthalate            | 84-66-2    | 5   |
| 36.           | 4-Chlorophenyl-phenylether  | 7005-72-3  | 5   |
| 37.           | Fluorene                    | 86-73-7    | 5   |
| 38.           | 4-Nitroaniline              | 100-01-6   | 20  |
| 39.           | 4,6-Dinitro-2-methylphenol  | 534-52-1   | 20  |
| 40.           | N-Nitrosodiphenylamine      | 86-30-6    | 5   |
| 41.           | 4-Bromophenyl-phenylether   | 101-55-3   | 5   |
| 42.           | Hexachlorobenzene           | 118-74-1   | 5   |
| 43.           | Pentachlorophenol           | 87-86-5    | 20  |
| 44.           | Phenanthrene                | 85-01-8    | 5   |
| 45.           | Anthracene                  | 120-12-7   | 5   |
| 46.           | Di-n-butylphthalate         | 84-74-2    | 5   |
| 47.           | Fluoranthene                | 206-44-0   | 5   |
| 48.           | Pyrene                      | 129-00-0   | 5   |
| 49.           | Butylbenzylphthalate        | 85-68-7    | 5   |
| 50.           | 3,3'-Dichlorobenzidine      | 91-94-1    | 5   |
| 51.           | Benz(a)anthracene           | 56-55-3    | 5   |
| 52.           | Chrysene                    | 218-01-9   | 5   |
| 53.           | bis-(2-Ethylhexyl)phthalate | 117-81-7   | 5   |
| 54.           | Di-n-octylphthalate         | 117-84-0   | 5   |
| 55.           | Benzo(b)fluoranthene        | 205-99-2   | 5   |
| 56.           | Benzo(k)fluoranthene        | 207-08-9   | 5   |
| 57.           | Benzo(a)pyrene              | 50-32-8    | 5   |
| 58.           | Indeno(1,2,3-cd)pyrene      | 193-39-5   | 5   |
| 59.           | Dibenz(a,h)anthracene       | 53-70-3    | 5   |
| 60.           | Benzo(g,h,i)perylene        | 191-24-2   | 5   |

<sup>1</sup>CRQLs are based on the CLP Organics Low Concentration SOW

TABLE 2  
ORGANIC COMPOUND  
SPECIFICATIONS  
(Continued)

| Pesticides/PCBs |                     | CAS Number | Contract Required<br>Quantitation Limits <sup>1</sup><br>(µg/L) |
|-----------------|---------------------|------------|---|
| 1.              | alpha-BHC           | 319-84-6   | 0.01  |
| 2.              | beta-BHC            | 319-85-7   | 0.01  |
| 3.              | delta-BHC           | 319-86-8   | 0.01  |
| 4.              | gamma-BHC (Lindane) | 58-89-9    | 0.01  |
| 5.              | Heptachlor          | 76-44-8    | 0.01  |
| 6.              | Aldrin              | 309-00-2   | 0.01  |
| 7.              | Heptachlor epoxide  | 1024-57-3  | 0.01  |
| 8.              | Endosulfan I        | 959-98-8   | 0.01  |
| 9.              | Dieldrin            | 60-57-1    | 0.02  |
| 10.             | 4,4'-DDE            | 72-55-9    | 0.02  |
| 11.             | Endrin              | 72-20-8    | 0.02  |
| 12.             | Endosulfan II       | 33213-65-9 | 0.02  |
| 13.             | 4,4'-DDD            | 72-54-8    | 0.02  |
| 14.             | Endosulfan sulfate  | 1031-07-8  | 0.02  |
| 15.             | 4,4'-DDT            | 50-29-3    | 0.02  |
| 16.             | Methoxychlor        | 72-43-5    | 0.10  |
| 17.             | Endrin ketone       | 53494-70-5 | 0.02  |
| 18.             | Endrin aldehyde     | 7421-36-3  | 0.02  |
| 19.             | alpha-Chlordane     | 5103-71-9  | 0.01  |
| 20.             | gamma-Chlordane     | 5103-74-2  | 0.01  |
| 21.             | Toxaphene           | 8001-35-2  | 1.0   |
| 22.             | Aroclor-1016        | 12674-11-2 | 0.20  |
| 23.             | Aroclor-1221        | 11104-28-2 | 0.20  |
| 24.             | Aroclor-1232        | 11141-16-5 | 0.40  |
| 25.             | Aroclor-1242        | 53469-21-9 | 0.20  |
| 26.             | Aroclor-1248        | 12672-29-6 | 0.20  |
| 27.             | Aroclor-1254        | 11097-69-1 | 0.20  |
| 28.             | Aroclor-1260        | 11096-82-5 | 0.20  |

<sup>1</sup>CRQLs are based on the CLP Organics Low Concentration SOW

### SECTION III

#### SAMPLE CONTAINER PREPARATION AND CLEANING PROCEDURES

This Section is provided as guidance for the preparation of sample containers that meet the contaminant-free specifications contained in Section II. There are various procedures for cleaning sample containers depending upon the analyses to be performed on the sample. The following cleaning procedures are modeled after those specified for the Superfund Sample Container Repository program. Other suitable cleaning procedures exist and may be used as long as the sample containers meet the criteria established in Section II. In some instances, the specific needs of the bottle user may dictate exact adherence to the sample container preparation and cleaning procedures that follow; while in other instances, modifications may be required. It is the responsibility of the bottle user to define the sample container preparation, cleaning, and quality control requirements.

- A. Cleaning Procedure for Container Types: A, E, F, G, H, J, and K
  - 1. Sample Type: Semivolatile Organics, Pesticides, Metals, Cyanide, and Fluoride in Soils and Water.
    - a. Wash glass bottles, Teflon liners, and caps with hot tap water using laboratory grade nonphosphate detergent.
    - b. Rinse three times with copious amounts of tap water to remove detergent.
    - c. Rinse with 1:1 nitric acid (reagent grade  $\text{HNO}_3$ , diluted with ASTM Type I deionized water).
    - d. Rinse three times with ASTM Type I organic free water.
    - e. Oven dry bottles, liners, and caps at 105-125°C for one hour.
    - f. Allow bottles, liners, and caps to cool to room temperature in an enclosed contaminant-free environment.
    - g. Rinse bottles with pesticide grade methylene chloride (or other suitable solvents specified by the bottle user) using 20 mL for 4-gallon containers; 10 mL for 12-oz and 16-oz containers; and 5 mL for 8-oz and 4-oz containers.
    - h. Oven dry bottles, liners, and caps at 105-125°C for one hour.
    - i. Allow bottles, liners, and caps to cool to room temperature in an enclosed contaminant-free environment.
    - j. Place liners in lids and cap containers.



- k. Label each container with the lot number and pack in a case.
  - l. Label exterior of each case with the lot number.
  - m. Store in a contaminant-free area.
2. Sample Type: Nitrate/Nitrite in Soils and Water.
- a. Substitute reagent grade sulfuric acid ( $H_2SO_4$ ) for nitric acid in step A.1.c.
  - b. Follow all other steps in the cleaning procedure described in part A.1 above.
- B. Cleaning Procedure for Container Types: B, D
1. Sample Type: Purgeable (Volatile) Organics in Soils and Water.
- a. Wash glass vials, Teflon-backed septa, Teflon liners, and caps in hot water using laboratory grade nonphosphate detergent.
  - b. Rinse three times with copious amounts of tap water to remove detergent.
  - c. Rinse three times with ASTM Type I organic-free water.
  - d. Oven dry vials, caps, septa, and liners at 105-125°C for one hour.
  - e. Allow vials, caps, septa, and liners to cool to room temperature in an enclosed contaminant-free environment.
  - f. Seal 40-mL vials with septa (Teflon side down) and cap.
  - g. Place liners in lids and cap 120-mL vials.
  - h. Label each vial with the lot number and pack in a case.
  - i. Label exterior of each case with the lot number.
  - j. Store in a contaminant-free area.
- C. Cleaning Procedure for Container Types: C, L
1. Sample Type: Metals, Cyanide, and Fluoride in Soils and Water.
- a. Wash polyethylene bottles and caps in hot tap water using laboratory-grade nonphosphate detergent.
  - b. Rinse three times with copious amounts of tap water to remove detergent.

- c. Rinse with 1:1 nitric acid (reagent grade  $\text{HNO}_3$ , diluted with ASTM Type I deionized water).
  - d. Rinse three times with ASTM Type I deionized water.
  - e. Invert and air dry in a contaminant-free environment.
  - f. Cap bottles.
  - g. Label each container with the lot number and pack in a case.
  - h. Label exterior of each case with the lot number.
  - i. Store in a contaminant-free area.
2. Sample Type: Nitrate/Nitrite in Soils and Water.
- a. Substitute reagent grade sulfuric acid ( $\text{H}_2\text{SO}_4$ ) for nitric acid in step C.1.c.
  - b. Follow all other steps in the cleaning procedure described in part C.1 above.

## SECTION IV

### SAMPLE CONTAINER QUALITY ASSURANCE AND QUALITY CONTROL REQUIREMENTS

#### A. Quality Assurance

The objectives of this Section are to: (1) present procedures for evaluating quality assurance (QA) information to ensure that specifications identified in Section II have been met; and (2) discuss techniques for the quality control (QC) analysis of sample containers to be used in conjunction with the cleaning procedures contained in Section III.

The bottle vendor should establish a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements, production procedures, and tracking systems. The QAP should incorporate procedures for the inspection of incoming raw materials; preparation, cleaning, and labeling of container lots; quality control analyses of cleaned container lots; document control, including all documentation required for analysis, packing, shipping, and tracking of container lots; any necessary corrective actions; and any quality assessment measures implemented by management to ensure acceptable performance. The QAP should be available and provided to the bottle purchaser upon request.

Major QA/QC activities should include the inspection of all incoming materials, QC analysis of cleaned lots of containers, and monitoring of the container storage area. Complete documentation of all QC inspection results (acknowledging acceptance or rejection) should be kept as part of the permanent bottle preparation files. QA/QC records (e.g., preparation/QC logs, analytical data, data tapes, storage log) also should be stored in a central location within the facility.

Documentation indicating that the container lot has passed all QA/QC requirements should be provided by the bottle vendor to the bottle purchaser with each container lot. Documentation should include a signed and dated cover statement affirming that all QA/QC criteria were met. Copies of raw data from applicable analyses of the QC containers, laboratory standards, check samples, and blanks should be available and provided upon request. Original documentation should be retained for at least 10 years. Minimum documentation that should be available, if applicable, for each lot of containers includes:

- A statement that "Sample container lot \_\_\_\_\_ meets or exceeds all QA/QC criteria established in 'Specifications and Guidance for Contaminant-Free Sample Containers;'"
- Reconstructed Ion Chromatographs (RICs) from volatile and semivolatile organics determinations, including calibration verification standards, check samples, and blanks;
- GC chromatographs from pesticides determinations, including calibration verification standards, check samples, and blanks;

- ICP, hydride-ICP, or ICP-MS instrument readouts from metals determinations, including calibration verification standards, check samples, and blanks;
- AA raw data sheets and instrument readouts from metals determinations, including calibration verification standards, check samples, and blanks; and
- Cyanide, fluoride, and nitrate/nitrite raw data sheets and instrument readouts from these determinations, including calibration verification standards, check samples, and blanks.

Prior to the first shipment of containers, and at least annually thereafter, the bottle vendor should demonstrate its ability to meet the CRDLs and CRQLs, and establish the reproducibility of the cleaning techniques for each bottle type. The ability to meet the CRDLs and CRQLs is accomplished through the determination of instrument detection limits (IDLs). The bottle vendor should use the procedures in the current CLP Low Concentration Inorganics and Organics SOWs to determine IDLs. IDLs should be below the CRDLs or CRQLs. To establish the reproducibility for each bottle type, the bottle vendor should randomly pick seven containers from a cleaned lot and analyze as described in the Quality Control Analysis part of this Section. Parameter concentrations should be at or below the CRDL or CRQL for each bottle type. Documentation from these analyses should be available and provided upon request.

#### 1. Incoming Materials Inspection:

A representative item from each case of containers should be checked for conformance with specifications provided in Section II. Any deviation should be considered unacceptable. A log of incoming shipments should be maintained to identify material type, purchase order number, and delivery date. The date of incoming inspection and acceptance or rejection of the material should also be recorded on this log.

#### 2. Quality Control Inspection of Cleaned Lots of Containers:

Following container cleaning and labeling, containers should be randomly selected from each container lot to be used for QC purposes. The two categories of QC containers should be as follows:

##### a. Analysis QC Containers:

One percent of the total number of containers in each lot should be designated as the analysis QC container(s). For lots of less than 100 containers, one container should be designated as the analysis QC container. The sample container preparer should analyze the analysis QC container(s) to check for contamination prior to releasing the associated container lot for shipment. The QC analyses procedures specified in the Quality Control Analysis part of this Section for determining the presence of semivolatile and volatile organics, pesticides, metals, cyanide, fluoride, and nitrate/nitrite should be utilized.

For each analysis QC container(s), an appropriate QC number should be assigned that cross-references the QC container to the related lot of containers. For example, the QC number could be a seven-digit number sequentially assigned to each lot that has undergone QC analysis. Under this numbering scheme, the first alphabetical character would be the container type letter from Figure 1, the next four digits would be assigned sequentially in numerical order starting with "0001" for the first lot to undergo QC analyses, the sixth character would indicate the number of QC container for the lot, (e.g., "1" for the first QC container in the lot, "2" for the second, etc.) and the last character would be either a "C" to indicate clearance or an "R" to indicate rejection.

If the representative analysis QC container(s) passes QC inspection, the related lot of containers should be released, and the appropriate QC number should be entered in the preparation/QC log to indicate clearance of the lot for shipment.

If the analysis QC container(s) are found to be contaminated per the specified QC analysis procedures, the appropriate QC rejection number should be assigned and entered in the preparation/QC log. Any container labels should be removed and the entire lot returned for reprocessing under a new lot number. Excessive QC rejection for a particular container type should be noted for future reference.

A laboratory standard, check sample, and a blank should be run with each QC analysis. A calibration verification standard should be analyzed once every 12 hours. All QC analysis results should be kept in chronological order by QC report number in a central QC file. The QC numbers assigned should be documented in the preparation/QC log, indicating acceptance or rejection and date of analysis.

A container lot should not be released for shipment prior to QC analysis and clearance. Once the containers have passed QC inspection, the containers should be stored in a contaminant-free area until packaging and shipment.

#### **b. Storage QC Containers:**

One QC container per lot should be designated as the storage QC container. The storage QC container should be separated from the lot after cleaning and labeling and should be stored in a designated contaminant-free area for one year. The date the container is placed in the storage area should be recorded in the storage QC container log.

If contamination of the particular container lot comes into question at any time following shipment, the storage QC container should be removed from the storage area and analyzed using the QC analysis procedures for that container type (see Quality Control Analysis, this Section). Upon removal, containers should be logged out of the storage area.

The designated storage area should be monitored continuously for volatile contaminants in the following manner. A precleaned, 40-mL via.

that has passed a QC inspection should be filled with ASTM Type I organic-free water and be placed in the storage area. This vial should be changed at one-week intervals. The removed vial should be subjected to analysis for volatile organics as described in the Quality Control Analysis part of this Section. Any peaks indicate contamination. Identify contaminants, if present, and include the results in a report to all clients who purchased bottles from the affected lot(s).

### **3. Quality Control Analysis**

The types of QC analyses correlate with the types of containers being analyzed and their future use in sample collection. The QC analyses are intended for the determination of:

- Semivolatile organics and pesticides;
- Volatile organics;
- Metals;
- Cyanide;
- Fluoride; and
- Nitrate/Nitrite.

QC analyses should be performed according to the container type and related sample type and utilize the specific method(s) described below.

#### **1. Determination of Semivolatile Organics and Pesticides:**

Container Types: A, E, F, G, H, J, and K

##### **a. Sample Preparation:**

- Add 60 mL of pesticide-grade methylene chloride to the container and shake for two minutes.
- Transfer the solvent to a Kuderna-Danish (KD) apparatus equipped with a three-ball Snyder column. Concentrate to less than 10 mL on a steam bath. Split the solvent into two 5 mL fractions for semivolatile and pesticide determinations.
- Add 50 mL of pesticide-grade hexane (for pesticide determinations only) to the KD apparatus by slowly pouring down through the Snyder column. Concentrate to less than 10 mL to effect solvent replacement of hexane for methylene chloride.
- Concentrate the solvent to 1 mL using a micro-Snyder column.
- Prepare a solvent blank by adding 60 mL of the rinse solvent used in step "g" of the cleaning procedure for container types A, E,

F, G, H, J, and K (Section III page 15) directly to a KD apparatus, and proceed as above.

**b. Semivolatile Organics Sample Analysis:**

- Instrument calibration should be performed as described in the most recent CLP Low Concentration Organics SOW with the following exceptions:
  - (1) If problems are encountered meeting the IRSD criteria on the initial calibration for semivolatiles, the high concentration point should be deleted and a four-point calibration used.
  - (2) The low concentration standard should be used for the continuing calibration standard for semivolatile analyses.
  - (3) The percent difference window should be widened to  $\pm 10$  percent for all compounds.
- Inject 1  $\mu$ L of solvent into a gas chromatograph/mass spectrometer (GC/MS).
- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Organics SOW.
- Blanks should be run as described in the most recent CLP Low Concentration Organics SOW.
- If peaks are found in the container blank that are not in the solvent blank, or if the container blank peak heights or areas are greater than 50 percent of the solvent blank peak heights or areas, the containers should be rejected.
- Identify and quantitate any contaminant(s) that cause rejection of a container lot.
- A standard mixture of the nine semivolatile organic compounds listed in Table 3 (page 29) with concentrations in the 5-20 ppb range should be analyzed to ensure that sensitivities are achieved that will meet contract required quantitation limits. This standard should be prepared from a different source from the calibration standards.

**c. Pesticides Sample Analysis:**

- Instrument calibration should be performed as described in the most recent CLP Low Concentration Organics SOW.
- Inject 1  $\mu$ L of solvent into a gas chromatograph (GC) equipped with an electron capture detector (ECD).
- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Organics SOW.

- Blanks should be run as described in the most recent CLP Low Concentration Organics SOW.
- If peaks are found in the container blank that are not in the solvent blank, or if the container blank peak heights or areas are greater than 50 percent of the solvent blank peak heights or areas, the containers should be rejected.
- Identify and quantitate any contaminant(s) that cause rejection of a container lot.
- A standard mixture of the seven pesticide compounds listed in Table 3 (page 29) with concentrations in the 0.01 to 1 ppb range should be analyzed to ensure that sensitivities are achieved that will meet contract required quantitation limits. This standard should be prepared from a different source from the calibration standards.

## 2. Determination of Volatile Organics:

Container Types: B and D

### a. Sample Preparation:

- Fill the container with ASTM Type I organic-free water.
- Cap the container and let stand for 48 hours.

### b. Sample Analysis:

- Instrument calibration should be performed as described in the most recent CLP Low Concentration Organics SOW with the following exceptions:
  - (1) If problems are encountered meeting the VRSD criteria on the initial calibration for volatiles, the high concentration point should be deleted and a four-point calibration used.
  - (2) The low concentration standard should be used for the continuing calibration standard for volatile analyses.
  - (3) The percent difference window should be widened to  $\pm 20$  percent.
- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Organics SOW.
- Blanks should be run as described in the most recent CLP Low Concentration Organics SOW. The blank should consist of an aliquot of the ASTM Type I water used in the sample preparation.
- If peaks are found in the container blank that are not in the solvent blank, or if the container blank peak heights or areas



are greater than 50 percent of the solvent blank peak heights or areas, the containers should be rejected.

- Identify and quantitate any contaminant(s) that cause rejection of a container lot.
- A standard mixture of the five volatile organic compounds listed in Table 3 (page 29) with concentrations in the 1-5 ppb range should be analyzed to ensure that sensitivities are achieved that will meet contract required quantitation limits. This standard should be prepared from a different source from the calibration standards.

### 3. Determination of Metals:

Container Types: A, C, E, F, G, H, J, K and L

#### a. Sample Preparation:

- Add 100 mL of ASTM Type I deionized water to the container, and acidify with 1.0 mL of reagent-grade  $\text{HNO}_3$ . Cap and shake for three to five minutes.
- Cap the container and let stand for 48 hours.
- Treat the sample as a dissolved metals sample. Analyze the undigested water using the most recent CLP Low Concentration Inorganics SOW.

#### b. Sample Analysis:

- Instruments used for the analysis of the samples should meet the contract required detection limits in Table 1.
- The ASTM Type I deionized water should be analyzed before use on the bottles that are designated for analysis to ensure that contaminated water is not used for rinsing the bottles.
- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW.
- Blanks should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW. A calibration blank is a solution made up exactly like the sample preparation solution. The calibration blank should be less than the values contained in Table 1.
- A set of standards in the expected working range should be analyzed with each analytical run. The acid matrix of the standards, blank, and quality control samples should match that of the samples.

- Concentrations at or above the detection limit for each parameter (listed in Table 1) should be cause for rejection of the lot of containers. **NOTE:** The sodium detection limit for container types A, E, F, G, H, J, and K is 5000 µg/L unless the containers will be used for low concentration analyses, then the detection limit is 500 µg/L.

4. **Determination of Cyanide:**

Container Types: A, C, E, F, G, H, J, K and L

a. **Sample Preparation:**

- Place 250 mL of ASTM Type I deionized water in the container. Add 1.25 mL of 6N NaOH (for container types F and G use 100 mL of ASTM Type I deionized water and 0.5 mL of 6N NaOH). Cap the container and shake vigorously for two minutes.

b. **Sample Analysis:**

- Analyze an aliquot as described in the most recent CLP Low Concentration Inorganics SOW.
- The detection limit should be 10 µg/L or lower.
- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW.
- Blanks should be run as described in the most recent CLP Low Concentration Inorganics SOW. The calibration blank should consist of an aliquot of the ASTM Type I water used above.
- A set of standards in the expected working range, a check sample, and blank should be prepared exactly as the sample was prepared.
- The detection of 10 µg/L cyanide (or greater) should be cause for rejection of the lot of containers. **NOTE:** Contamination could be due to the container, the cap, or the NaOH.

5. **Determination of Fluoride:**

Container Types: A, C, E, F, G, H, J, K and L

a. **Sample Preparation:**

- Place 250 mL of ASTM Type I deionized water in the container (for container types F and G use 100 mL of ASTM Type I deionized water). Cap the container and shake vigorously for two minutes.

b. **Sample Analysis:**

- Analyze an aliquot as described in the most recent CLP Low Concentration Inorganics SOW.

- The detection limit should be 200 µg/L or lower.
- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW.
- Blanks should be run as described in the most recent CLP Low Concentration Inorganics SOW. The calibration blank should consist of an aliquot of the ASTM Type I water used above.
- A set of standards in the expected working range, a check sample, and blank should be prepared exactly as the sample was prepared.
- The detection of 200 µg/L (or greater) of fluoride should be cause for rejection of the lot of containers. **NOTE:** Contamination could be due to the container or the cap.

6. **Determination of Nitrate/Nitrite:**

Container Types: A, C, E, F, G, H, J, K and L

a. **Sample Preparation:**

- Place 250 mL of ASTM Type I deionized water in the container (for container types F and G use 100 mL of ASTM Type I deionized water). Cap the container and shake vigorously for two minutes.

b. **Sample Analysis:**

- Analyze an aliquot as described in the most recent CLP Low Concentration Inorganics SOW.
- The detection limit should be 100 µg/L or lower.
- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW.
- Blanks should be run as described in the most recent CLP Low Concentration Inorganics SOW. The calibration blank should consist of an aliquot of the ASTM Type I water used above.
- A set of standards in the expected working range, a quality control sample, and blank should be prepared exactly as the sample was prepared.
- The detection of 100 µg/L (or greater) of nitrate/nitrite should be cause for rejection of the lot of containers. **NOTE:** Contamination could be due to the container or the cap.

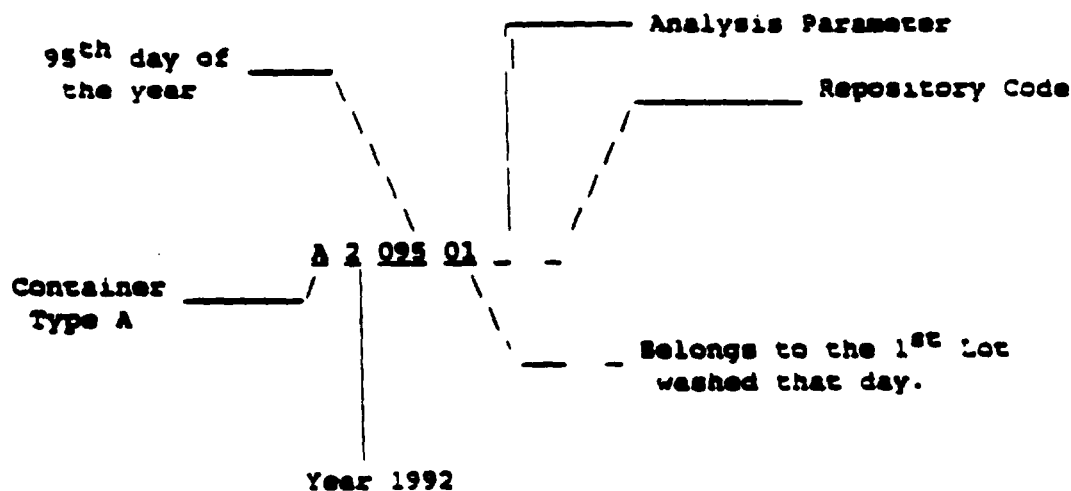
### C. Preparation and Labeling

Sampling for environmental specimens requires that sample containers be transported to field sites prior to sample collection. As a result, considerable time may elapse between the receipt of sample containers and collection of the samples. Because of the large number of samples taken at any one site, accounting for all sample containers can become extremely difficult. The following guidance on the identification and tracking of sample containers is based on procedures that have been used successfully in the CLP bottle program.

1. Each shipment should be inspected to verify that the requested number of cleaned and prepared sample containers have been supplied and meet the requirements specified in Section II (Tables 1 and 2). If any shipment fails to meet the required specifications, it should be discarded and replaced with a supply of sample containers that meet the required criteria.
2. The sample containers should be removed and prepared in accordance with the methods designated below.
3. A permanent nine-digit lot number should be assigned to each lot of sample containers for identification and tracking purposes throughout the life of the containers. Figure 2 provides an example of a lot number sequence.

FIGURE 2

#### LOT NUMBER SEQUENCE



- a. The first digit represents the container type in Section II (Figure 1).
- b. The second digit represents the last digit of the calendar year.

- c. The next three digits represents the day of the year on which the sample containers were washed.
  - d. The sixth and seventh digits represent the daily lot number.
  - e. The eighth digit represents the analysis parameter where:
    - A = Semivolatile organics, pesticides, metals, cyanide, and fluoride;
    - B = Metals, cyanide, and fluoride;
    - V = Volatile organics;
    - S = Semivolatile organics and/or pesticides;
    - M = Metals;
    - C = Cyanide;
    - F = Fluoride; and
    - N = Nitrate/nitrite.
  - f. The final digit represents the identification of the person who prepared the lot.
4. The lot number for each container should be entered, along with the date of washing, type of container, and number of containers per lot, into the preparation/QC log book.
  5. Lot numbers printed with solvent resistant ink on a nonremovable label should remain with the corresponding containers throughout the cleaning procedure.
  6. After sample container cleaning and drying, the label should be affixed to the containers in a permanent manner.
  7. At least one face should be clearly marked, excluding the top and bottom faces, of each case of sample containers with the assigned lot numbers.

**TABLE 3**

**STANDARD MIXTURES OF ORGANIC COMPOUNDS TO VERIFY SENSITIVITY**

| <b>Volatiles</b>   | <b>Semivolatiles</b>       | <b>Pesticides</b> |
|--------------------|----------------------------|-------------------|
| Methylene Chloride | Nitrobenzene               | Gamma-BHC         |
| Acetone            | 4-Chloroaniline            | Heptachlor        |
| 2-Butanone         | 2,6-Dinitrotoluene         | Aldrin            |
| Trichloroethene    | Diethylphthalate           | Dieldrin          |
| Toluene            | 4-Bromophenyl-phenylether  | Endrin            |
|                    | Hexachlorobenzene          | 4,4'-DDT          |
|                    | Pentachlorophenol          | Aroclor 1260      |
|                    | Di-n-butylphthalate        |                   |
|                    | bis(2-Ethylhexyl)phthalate |                   |